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FRAC TOGRAPHIC AND MICROSTRUCTURAL ANALYSIS OF STRESS CORROSION --ETC(U)

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**Fractographic and Microstructural Analysis of Stress  
Corrosion Cracking of A533 Grade B Class 1 Plate and  
A508 Class 2 Forging in Pressurized Reactor-Grade  
Water at 93°C**

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)  Stress corrosion cracking (SCC) studies were conducted in two commonly used pressure vessel steels: A533 Grade B Class 1 (A533-B-1) plate and an equivalent A508 Class 2 (A508-2) forging. The purpose of these studies was to determine the response of the materials in simulated pressurized water reactor environment. Round tensile specimens, 32 mm (1.25 in.) along the gage section and 9.5 mm (0.375 in.) in diameter, were bolt-loaded to 75 to 80 percent of yield and left in the water at 93°C for 2000 hr. At the end of this time period, the specimens were taken out of the water, (Continues)		

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## 20. Abstract (Continued)

unloaded, and examined by scanning electron microscopy (SEM) and by energy dispersive x-ray analysis. The specimen cut from A533-B-1 plate did not develop any cracks as a result of the SCC tests. Several cracks, some of which were visible with the unaided eye, had developed in the specimen that was cut from the A508-2 forging. These cracks ran along the gage section and were perpendicular to the direction of the applied stress. The largest crack was about 9 mm in circumferential length and 3 mm in depth. After visual examination, the specimens were cut lengthwise and sectioned for the microstructural studies. The polished and etched sections of the specimen that showed SCC when examined with the SEM revealed the presence of many microcracks starting from the outside surface and propagating either along inclusion-matrix or carbide-matrix interfaces; its corresponding microstructure was mostly pearlitic. The microstructure of the specimen that did not crack was bainitic, had fewer inclusions and carbide particles and no microcracks were observed. When the fracture surfaces of the larger cracks were examined with the SEM, they revealed a cleavage-like failure propagating along inclusion bands. X-ray image scans and energy spectra of these bands showed that they were silicate and manganese-sulfide inclusions.

A hydrogen-assisted cracking model has been proposed to explain the experimental results on A508-2 forging. Produced by a cathodic reaction and aided by the stress fields, hydrogen diffuses ahead of the crack tip to the inclusion sites. This causes a preferential decohesion at the inclusion matrix interfaces and subsequent cracking along inclusion bands.

The absence of stress corrosion cracking in A533-B-1 plate tested under identical experimental conditions is mainly due to fewer inclusion and carbide particles and to the more refined bainitic microstructure of this steel. This type of microstructure is less susceptible to hydrogen-assisted cracking than the mainly pearlitic microstructure found in A508-2 forging.

## CONTENTS

INTRODUCTION .....	1
MATERIALS AND TEST SPECIMENS .....	2
EXPERIMENTAL PROCEDURE .....	2
RESULTS AND DISCUSSION .....	4
ENVIRONMENTAL-ASSISTED CRACKING MODEL .....	13
SUMMARY AND CONCLUSIONS .....	13
ACKNOWLEDGMENTS .....	14
REFERENCES .....	15

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# FRACTOGRAPHIC AND MICROSTRUCTURAL ANALYSIS OF STRESS CORROSION CRACKING OF A533 GRADE B CLASS I PLATE AND A508 CLASS 2 FORGING IN PRESSURIZED REACTOR-GRADE WATER AT 93°C

## INTRODUCTION

Primary pressure vessels for water-cooled nuclear reactors are fabricated from low alloy steels such as A533 Grade B Class 1 plate and A508 Class 2 forging. The combinations of sustained and cyclic loads can give rise to the phenomena of stress corrosion cracking (SCC) and/or corrosion fatigue.<sup>a</sup> SCC is known to be a problem in high strength and the austenitic steels when exposed to certain aggressive environments [1-3]. Recently, however, limited data have become available on low alloy steels showing that an aqueous environment at temperatures close to 100°C can promote SCC in these materials [4,5]. Other data concern SCC failures of steam turbines in conventional power plants [6,7].

Lately, especially in connection with the light water reactor safety program, a great deal of attention has been given to the potential problems of corrosion fatigue occurring in the low alloy steels exposed to a pressurized water environment [8]. Little, however, has been done in the area of stress corrosion cracking occurring in these steels when exposed to similar environments. The present SCC study on A533-B-1 plate and A508-2 forging and the subsequent fractographic and microstructural analyses is being conducted to assess the magnitude of the problem. The study developed when a stress corrosion cracking component appeared to be present, under certain test conditions, in A508-2 forging fatigue tested in reactor-grade water at high temperatures [8].

In the present work, two bolt-loaded tensile specimens, one of A533-B-1 plate and the other of A508-2 forging, exposed to a reactor grade water at 93°C for 2000 hr, were examined by scanning electron microscopy (SEM), and by energy dispersive x-ray analysis. The basic aims of the fractographic and microstructural analyses were to search for correlations and relationships between the fractographic features and microstructural components on one hand, and the SCC behavior of the two steels on the other.

<sup>a</sup>Stress corrosion cracking is the cracking process that is caused by the joint action of a mechanical load and an aggressive environment, while by corrosion fatigue is meant the crack growth resulting from cyclic stresses and an aggressive environment.

Note: Manuscript submitted November 13, 1979.

## MATERIALS AND TEST SPECIMENS

The materials used in this investigation were A533 Grade B Class 1 plate and A508 Class 2 forging pressure vessel steels. The chemical compositions for both steels are given in Table 1. A533-B-1 plate, a high shelf, low sulfur content steel, was obtained from the Heavy Section Steel Technology (HSST) Program; the A508-2 forging, of foreign fabrication, came from a large 200-ton block intended for a flange ring for a reactor pressure vessel. The forging, however, had been rejected because non-destructive and metallographic examinations had revealed the presence of cracks at the center and macrosegregation, i.e., an excessive amount of inclusions throughout the forging block. The above two pressure vessel steels were chosen for the SCC tests because they constitute a sort of best case-worst case study. That is, the intent was to examine the SCC response in a simulated reactor-water environment at 93°C of materials of different qualities - one of relatively high quality (the A533-B-1 plate) and the other of relatively low quality (the A508-2 forging). The A508-2 tensile specimen used for the SCC tests was cut along the through-thickness direction, from a section of the forging which was free of cracks but had excessive segregation. Round tensile specimens were machined from both steels having the following dimensions: 32 mm (1.25 in.) along the gage section, and 9.5 mm (0.375 in.) in diameter. Prior to the SCC testing, the specimens were optically examined to be sure that they were free of machining flaws.

## EXPERIMENTAL PROCEDURE

The specimens were bolt-loaded, along the tensile axis, to about 85 to 90 percent of their respective yield strengths using a 53,400 N (12,000 lb) capacity tensile machine. The specimens were loaded in air at room temperature and the applied loads were measured by means of strain gages attached to the specimens. To avoid introducing large shear components on the specimens, the following loading procedure was employed. The specimen was loaded to 85 to 90 percent of its yield strength with the tensile machine and the strain gage reading was recorded for this stress level. Afterwards, the tensile load on the specimen was reduced to about 70 percent of yield. The bolts, on the loading assembly, were tightened while the specimen was still under tension. The specimen was taken out of the tensile machines when the bolt-loading produced a strain gage reading equivalent to 85 to 90 percent of the yield strength. To be sure that bolt-loading per se did not produce cracks in the specimens, following the first loading, the specimens were unloaded and examined once again with an optical microscope. Afterwards, the specimens were loaded a second time to the previous stress level (85 to 90 percent of the yield stress) using the same procedure described above. The fact that the loads were computed for, and applied at room temperature does not introduce any significant errors in the actual levels realized for the tests at the higher temperature. Further, the test jig was also made from A508, thus eliminating electrochemical reactions, and insuring that the (zero) differences in thermal expansion and elastic modulus did not produce any artificial load changes upon the change in temperature from 27 to 93°C. This was verified by the fact that the difference in the strain gage readings between room temperature and 93°C were the same in both the unstrained and strained condition.

Table 1 - Chemical composition of A508-2 forging and A533-B-1 plate

Alloy	Composition, Weight %																
	C	Si	Mn	P	S	Cu	Sn	Al	Ni	Cr	Mo	Ni	Co	Ta	As	Sb	Fe
A508-2	0.19	0.2	0.93	0.008	0.006	0.10	0.007	0.016	0.007	0.50	0.56	1.29	0.014	0.01	0.028	0.006	Bal.
A533-B-1	0.25	0.25	1.56	0.007	0.012	0.12	-	-	-	0.05	0.56	0.56	-	-	-	-	Bal.

Prior to the SCC tests, any stress relaxation both on the loading assembly and the specimen that might be caused by the temperature difference between the loading temperature (room temperature) and the test temperature (93°C) was measured by leaving the bolt-loaded specimens for a number of days inside an oven kept at 93°C. The specimens were taken out of the oven when it became apparent that all stress relaxation had stopped. The maximum stress relaxation measured this way was about 10 percent of the room temperature yield strength. That is, the final loading on the specimens was about 75 to 80 percent of the room temperature yield strength, which is, for all practical purposes, the same as the 93°C yield stress value.

The SCC tests were conducted using a closed environmental chamber known as the "water pot." This chamber is primarily used for fatigue studies on A508-2 pressure vessel steel in connection with the light water reactor safety program carried on at NRL for the U. S. Nuclear Regulatory Commission (NRC). The water pot holds about six liters of reactor grade water at 93°C at a pressure close to one atmosphere. The water in the chamber was circulated at about one liter per hour. The reactor-grade water used in the environmental chamber was high purity water containing 1000 ppm boron as boric acid and 1 ppm lithium as lithium hydroxide. Total impurity content of other elements was less than 1 ppm. The specific conductance of the reactor-grade water was 16 to 20  $\mu$ mhos/cm after exposure in the water pot, and the pH approached 5.8 from an initial value of 6.7 to 6.8. The dissolved oxygen level was held as low as possible (0 to 2 ppb) by continuous deoxygenation of the water. Additional details concerning this device may be found elsewhere [8]. The loaded specimens were placed in the environmental chamber and left there for 2000 hr. After this time period, the specimens were removed from the water pot, unloaded and examined by microscopic techniques.

The specimens were sectioned lengthwise for metallographic specimen preparation and subsequent microstructural analyses. One half of one specimen was broken in liquid nitrogen and the fracture area was examined with a scanning electron microscope and with an energy dispersive x-ray analyzer. The metallographic sections were ground and polished by conventional techniques. Prior to the polishing, the sections were nickel-plated for edge retention. The polished sections were etched in 1 percent nital solution for about one-half minute and then examined by SEM, using an ISI Super II instrument.

SEM examination of the metallographic sections was utilized to provide information on microstructural components. Phase identification was obtained by energy dispersive x-ray analysis employing an AMR 1000 SEM equipped with EDAX Model 707B dispersive energy x-ray analyzer. Fractographic examination was carried out using two different scanning electron microscopes: a Coates and Welter field emission microscope and an ISI Super II microscope. Both electron microscopes were operated at an accelerating voltage of 20 kV.

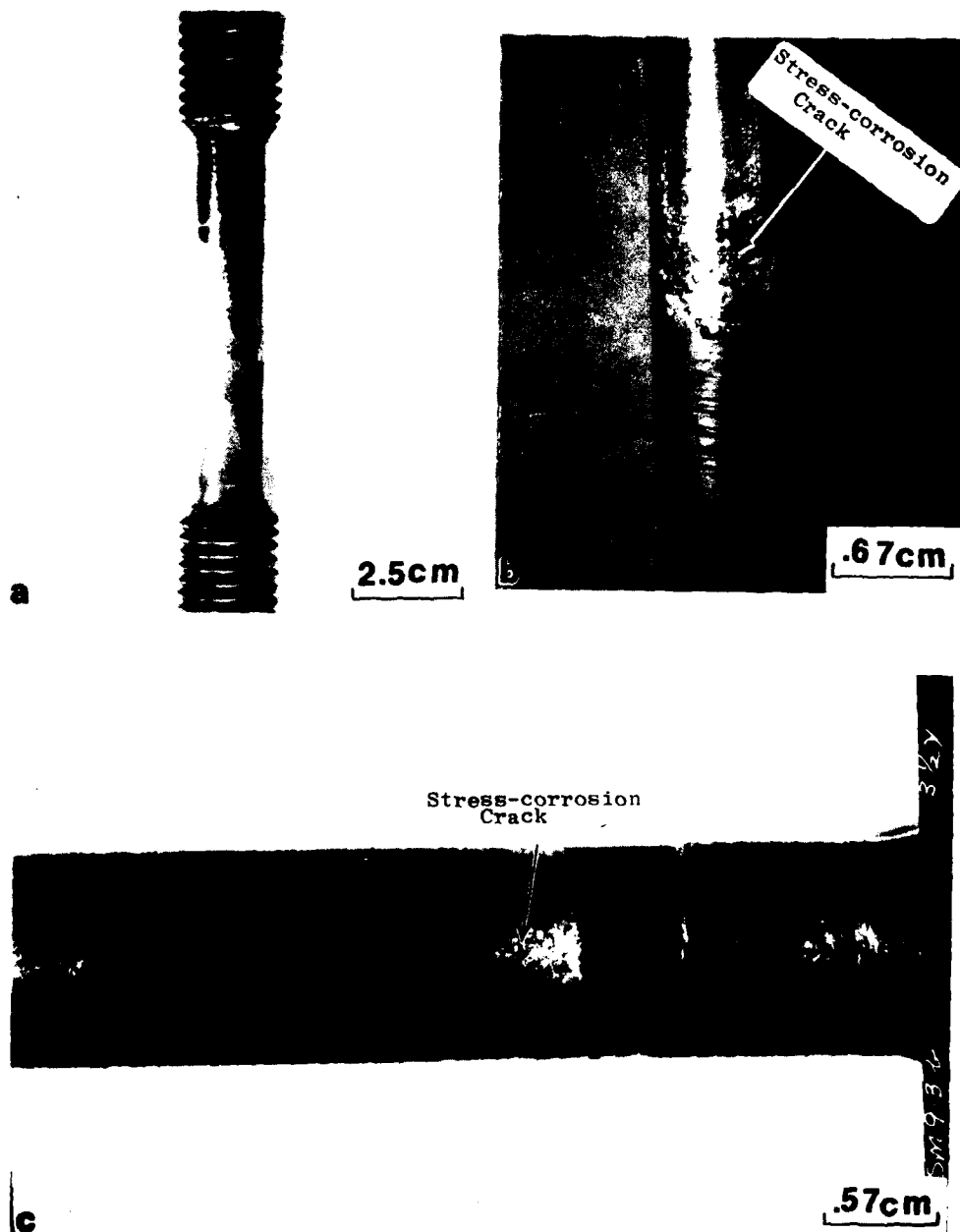
## RESULTS AND DISCUSSION

When the A508-2 specimen was taken out of the water pot and unloaded, seven different cracks were present along the specimen gage section. These

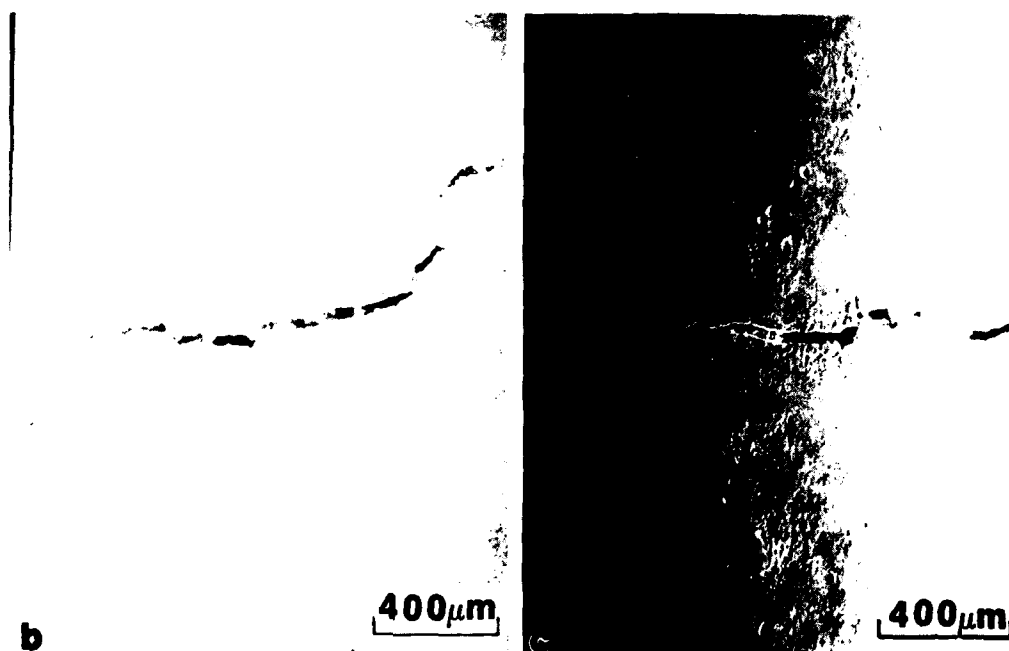
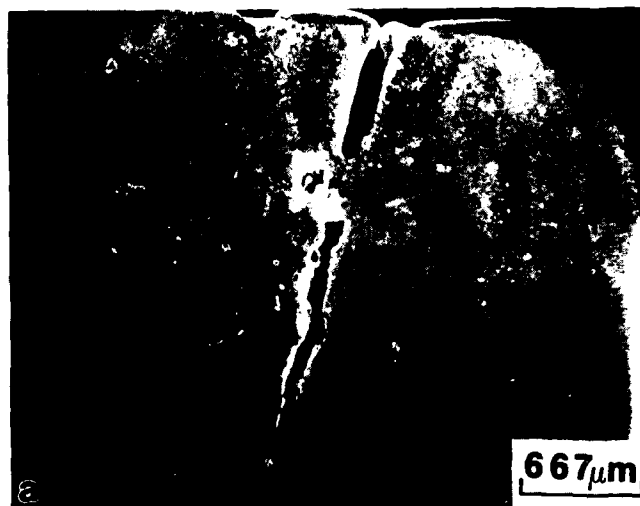
cracks were clearly visible with the unaided eye, and they ranged in circumferential length from 1.5 mm (0.060 in.) to 9 mm (0.360 in.); the depth of the largest crack was about 3 mm (0.12 in.). No cracks were seen in the A533-B-1 specimen which had been tested under identical experimental conditions. The photographs in Fig. 1 show the appearance of the A508 tensile specimen. These photographs show the entire specimen (Fig. 1a), the largest of the seven cracks (Fig. 1b), and a smaller crack (Fig. 1c). As can be seen from the photographs, the cracks are oriented perpendicular to the direction of the applied stress. Some other smaller cracks found also on the gage section of the A508-2 specimen are shown by the SEM micrographs in Fig. 2.

The SEM micrographs in Fig. 3 show typical features of the microstructure of A533-B-1 plate and A508-2 forging materials; these micrographs were taken from the polished and etched sections of the two specimens. The microstructure of the A508-2 forging (Fig. 3a) shows several inclusions and coarse carbides. On the other hand, the microstructure of the A533-B-1 plate (Fig. 3b) shows smaller carbides and fewer inclusion particles. An additional microstructural evaluation of the two steels showed that the microstructure of A508-2 forging was ferritic-pearlitic with small amounts of bainite, whereas that of A533-B-1 plate was completely bainitic. Figure 4 is a montage of SEM micrographs of a polished and etched section of A508-2 specimen showing one of the many microcracks that were present in this specimen. The microcrack started from the outside surface and propagated along the inclusion-matrix material interface. As can be seen in the micrographs, decohesion also occurred along the carbide-matrix interface. The SEM micrographs presented in Fig. 5 are higher magnification micrographs of the crack tip areas of the cracks shown in Fig. 1b and 2a and the microcrack in Fig. 4. Small voids are observed in the vicinity of the crack tip in both cases. These voids are similar to ones observed in a parallel study of the fatigue crack growth resistance of A508-2 forging steel in reactor-grade water at temperatures up to 288°C (550°F) [8]. In that study it was suggested that the small voids are the sites to which hydrogen can most easily diffuse and be trapped.

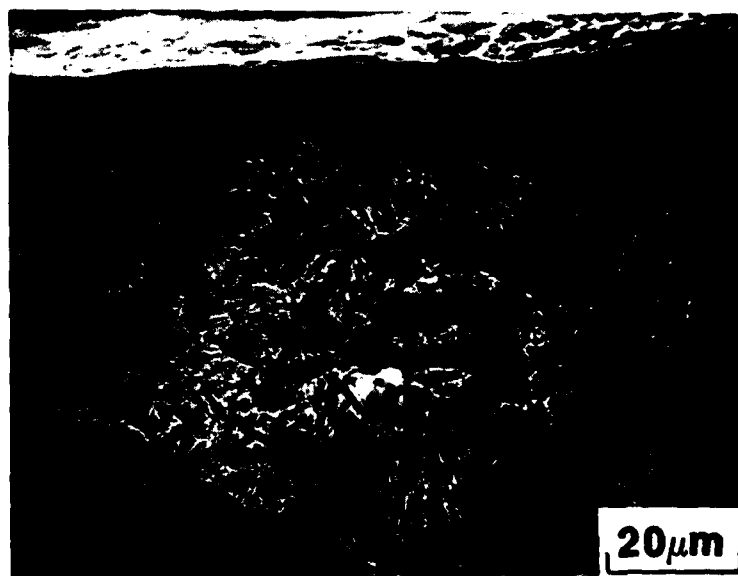
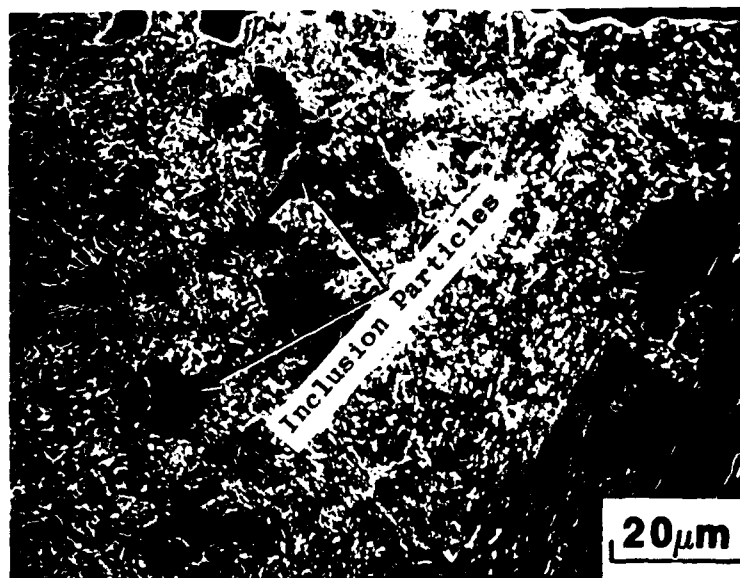
The significant fractographic features observed on the stress corrosion area in one of the several cracks found in the A508-2 specimen are illustrated in the SEM micrograph presented in Fig. 6. Figure 6a is a lower magnification micrograph that gives a general view of the stress corrosion area. The failure mode appears to be cleavage-like and inclusion particles are clearly visible on the fracture surface. The inclusions are quite evident in the adjacent higher magnification micrographs (Fig. 6b and c). The inclusion particles have an elongated shape and they are quite numerous. Panel (d) of the same figure is the energy dispersive x-ray spectrum of a small region in the stress corrosion area near an inclusion particle. The spectrum, besides the expected peaks of iron and chromium, reveal the presence of manganese, silicon and sulfur peaks and a small chlorine peak. The spectrum indicates that the inclusion particles are manganese-sulfide and silicate inclusions. The x-ray image scans presented in Fig. 7 refer to manganese and sulfur; similar image scans were obtained for the silicate inclusions.



**Fig. 1.** Optical photographs of A508-2 tensile specimens used in the SCC testing. Photograph (a) shows the entire specimen, photographs (b) and (c) show some of the larger cracks that developed on the gage section following exposure to reactor-grade water at  $93^{\circ}\text{C}$  for 2000 hr.



**Fig. 2.** SEM micrographs of gage and the transverse gage sections of A508-2 tensile specimen. Prior to SEM examination, the transverse section was metallographically polished and etched in 1 percent nital solution, the gage section was ultrasonically cleaned in alcohol and acetone. Fig. 2a shows the depth of the largest crack; Figs. 2b and c show two smaller cracks that developed during the SCC tests.



**Fig. 3.** SEM micrographs of A508-2 forging and of A533-B-1 plate specimens. The micrographs were taken from the transverse sections of the specimens. The microstructure of A508-2 (Fig. 3a) is characterized by large amounts of carbide and inclusion particles; the microstructure of A533-B-1 (Fig. 3b) has fewer carbide particles and it is almost free of inclusions.



**Fig. 4.** Montage of SEM micrographs of a transverse section of A508-2 specimen. The montage shows one of many microcracks found on the transverse sections after the SCC test. The microcrack originated at the outside surface and propagated along the inclusion-matrix and carbide-matrix interfaces.

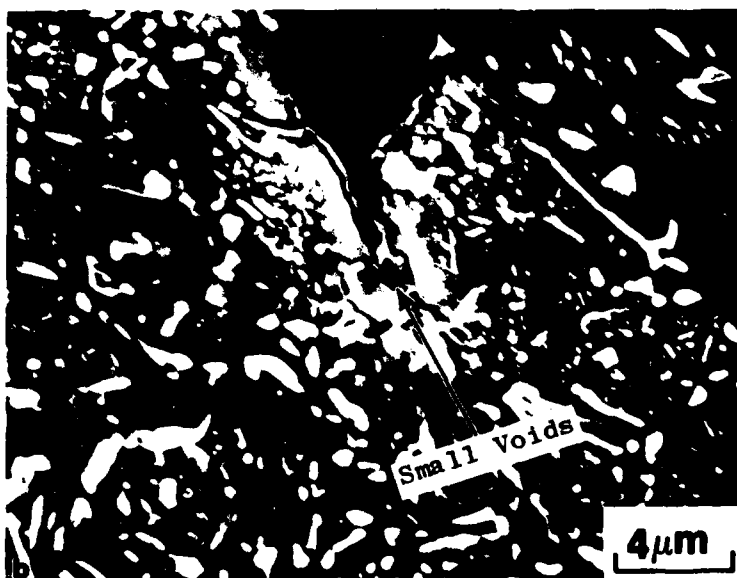
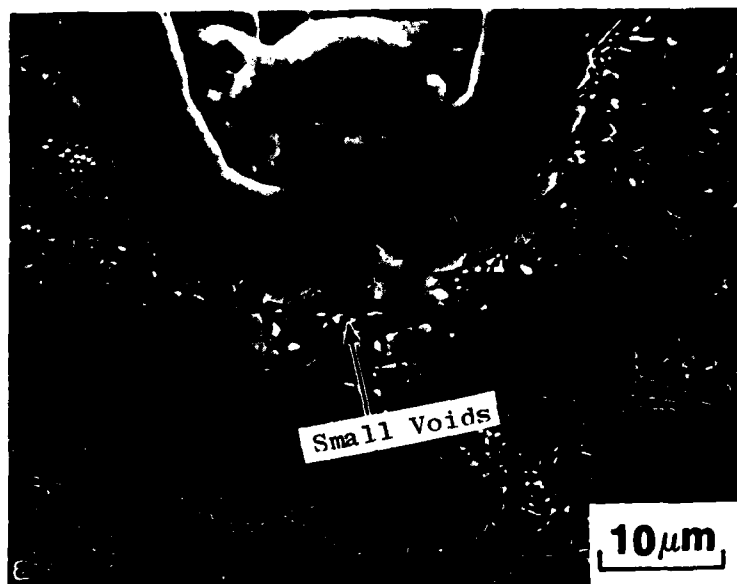


Fig. 5. SEM micrographs of transverse sections of A508-2 tensile specimen. Micrograph (a) is an enlarged view of the crack tip shown in Figs. 1b and 2a; micrograph (b) is an enlarged view of the tip of the microcrack shown in Fig. 4. The micrographs show the presence of small voids near the tips of both cracks.

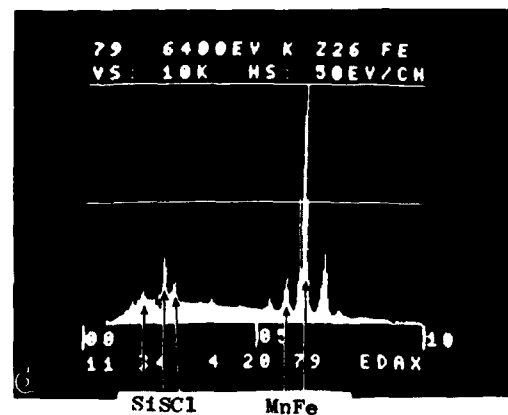
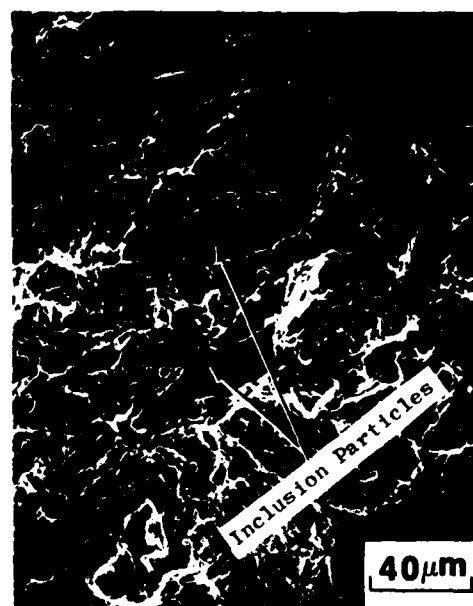
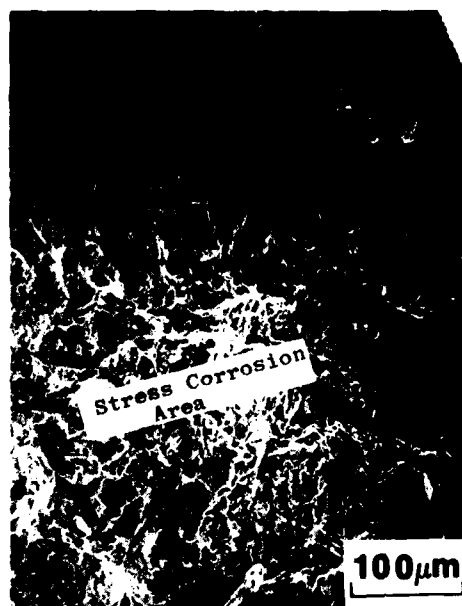


Fig. 6. SEM micrographs (a, b, and c), and energy dispersion x-ray spectrum (d) taken from the stress corrosion crack of a A508-2 tensile specimen. This stress corrosion crack became accessible for SEM examination when one half of the specimen was broken at liquid nitrogen temperature along one of the several cracks that had developed on the gage sections. Micrographs a through c show cleavage-like fracture propagation along inclusion bands. The x-ray spectrum shows that the bands are silicate and manganese-sulfide inclusions.

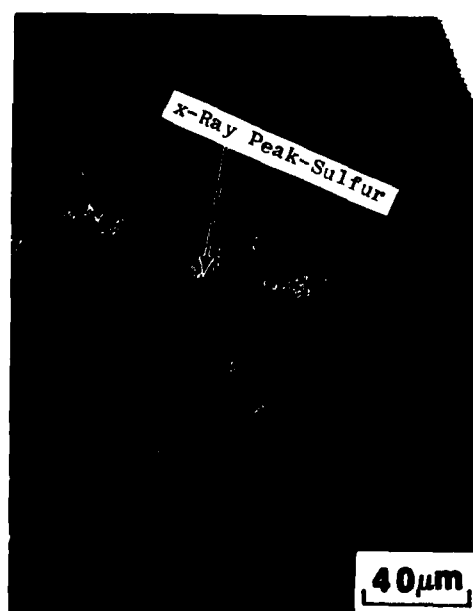
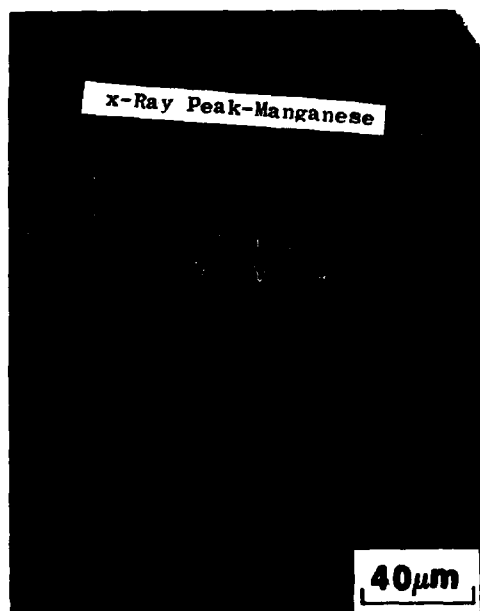
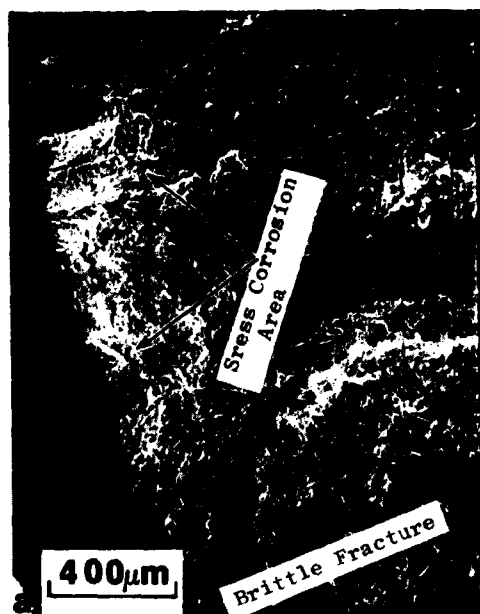


Fig. 7. Stress corrosion area (micrographs a and b) and corresponding energy dispersive x-ray image scans of A508-2 specimen. The stress corrosion area is the same one shown in the previous figure. The x-ray image scans refer to manganese and sulfur; similar image scans were obtained for the silicates.

To be sure that the SCC cracking observed on A508-2 forging was not the result of pure mechanical effects as, for example, creep cracking, a control test in a hydrogen environment was conducted on A508-2 forging. The same loading procedure and stress level used for the other two tests were employed in this control test. The bolt-loaded tensile specimen was exposed to pure hydrogen at ambient pressure for 2000 hr at 93°C. After this time the specimen was unloaded and examined. Optical examination showed that this specimen did not develop any cracks during SCC testing in gaseous hydrogen. This finding seems to suggest that SCC of A508-2 forging in reactor-grade water at 93°C is the result of an interaction between certain microstructural components and the aqueous environment.

#### ENVIRONMENTAL-ASSISTED CRACKING MODEL

A hydrogen-assisted cracking model has been developed to explain the above results. When the test specimen is exposed to a pressurized reactor-grade water at 93°C, an oxide layer is formed on the surface consisting mainly of hematite. Hematite is not protective, thus allowing further corrosion reactions to occur. Due to the presence of manganese-sulfide and silicate inclusions, the prevailing electrochemical conditions initiate a localized corrosion attack on the metal matrix around the inclusions [9,10] and a decrease of pH occurs [11,12]. Also the solubility of MnS even in pure water allows the formation of a sufficient concentration of HS<sup>-</sup> ions, which accelerates the corrosion reaction and hydrogen evolution [11,13]. Further enhancement of the corrosion attack may be caused by the migration of the chlorine ions into the crevice [9]. The energy dispersive x-ray spectrum shown in Fig. 6d provides support for this hypothesis.

Hydrogen is generated and subsequently absorbed into the metal due to the local cathodic reactions and hydrolysis of water. The absorbed hydrogen diffuses along the stress gradient produced by the propagating crack and the inclusions under the applied stress and hydrogen becomes trapped at the inclusions sites [13]. The presence of hydrogen at the interface lowers the bond strength between the inclusions and the matrix as well as the carbides causing the subsequent separation of the inclusions and the carbides from the matrix [14,15]. This is clearly illustrated in Figs. 4 and 5 which show voids around inclusions and carbides close to the crack tip. This sufficiently increases the local stress so that, with the assistance of hydrogen, crack propagation can occur in the metal matrix, as evidenced, by the cleavage-like features on the fracture surface. The absence of stress corrosion cracking in A533-B-1 plate steel tested under identical conditions is mainly due to smaller amounts of inclusions and also due to the more refined bainitic microstructure, which is less susceptible to hydrogen-assisted cracking than the mainly ferritic-pearlitic microstructure of A508-2 forging.

#### SUMMARY AND CONCLUSIONS

Fractographic and microstructural studies by scanning electron microscopy (SEM) and by energy dispersive x-ray have been conducted on two pressure vessel steels: A533-B-1 plate and A508-2 forging tested for stress corrosion cracking (SCC) in a reactor-grade water at 93°C. The specimens,

round tensile bars, bolt-loaded to about 75 to 80 percent of their respective yield strengths, were exposed to the water environment for 2000 hr. The significant observations from this study may be summarized as follows:

1. The specimen from the A508-2 forging developed several cracks along the gage section, some of which were clearly visible with the unaided eye. The specimen from the A533-B-1 plate did not develop cracks.
2. Examination of the polished and etched sections of both specimens revealed that the A508-2 forging had large amounts of inclusions and coarse carbide particles and its microstructure was mostly ferritic-pearlitic. In addition, many microcracks were observed that started from the outside surfaces and preferentially propagated either along the inclusion-matrix or along the carbide-matrix material interfaces. The microstructure of A533-B-1 plate was mostly bainitic and had much less carbide particles and was almost free of inclusions. Finally, no microcracks were seen in this specimen.
3. Subsequent x-ray scans of the inclusions showed them to be silicate and manganese-sulfide inclusions.
4. Fractographic examination of the stress corrosion area of one of the cracks found in the A508-2 specimen showed a cleavage-like failure with the crack propagating along inclusion bands.
5. A hydrogen embrittlement model has been proposed to explain the above results. For the specimen that cracked, hydrogen is produced at the crack by a cathodic reaction between the water environment and the fresh metal surface. It diffuses into the plastic enclave just ahead of the crack tip by following the stress gradient caused by the propagating crack and the inclusions and is trapped along the inclusion-matrix interfaces. The presence of hydrogen at the interfaces causes local embrittlement by lowering the bond strength between the inclusions and the matrix. On the other hand, the presence of much fewer inclusions and carbide particles in the A533-B-1 plate together with a more refined bainitic microstructure makes this material immune to stress corrosion cracking when exposed to reactor-grade water at 93°C.

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